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The crystal structure of (*Z*)-5,6-dihydro-8(7*H*)quinolinone thiosemicarbazone has been determined by X-ray single crystal analysis in order to confirm the identity of this analog of the antineoplastic agent 2-formylpyridine thiosemicarbazone. In the solid state, the molecules are arranged in pairs held together by intermolecular S-N hydrogen-bonds. There is also an extensive intramolecular N-N hydrogen-bonding system.

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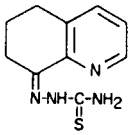
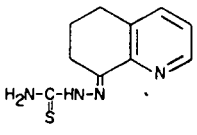
Sir:

It was observed that the reaction of thiosemicarbazide with 5,6-dihydro-8(7*H*)quinolinone yielded a mixture of (*Z*)-5,6-dihydro-8(7*H*)quinolinone thiosemicarbazone (**1**) and (*E*)-5,6-dihydro-8(7*H*)quinolinone thiosemicarbazone (**2**). The isomers were readily separated by column chromatography on silica gel using methanol:chloroform (1:19) as eluting solvent. The *Z* isomer, **1** (m.p. 187-190°) was eluted first followed by the *E* isomer, **2** (m.p. 184-185°). Because these compounds were synthesized as analogs of the antineoplastic agent 2-formylpyridine thiosemicarbazone (1), it was imperative that an unambiguous structure assignment be made. The *Z* and *E* configurations were tentatively assigned based on spectral considerations.



1



2

Crystals of the compound are monoclinic, space group $P2_1/n$, with a = 7.682(3), b = 10.168(8), c = 14.795(5) Å, β = 103.11(4)°, and Z = 4 formula weights per unit cell. Intensity data were collected at room temperature with an Enraf-Nonius CAD-4 diffractometer using Mo K$\bar{\alpha}$ (λ = 0.71073 Å) radiation monochromatized by a graphite crystal. A total of 1699 unique reflections in the range $6° < 2\theta < 45°$ were measured using the θ-2θ scan technique. The structure was solved by use of the program MULTAN (2). A final R value [$R = (\Sigma||F_o| - |F_c||)/\Sigma|F_o|$] of 0.017 was obtained by full-matrix least squares refinement of 1131 contributing reflections.

Figure 1 shows the bond distances between non-hydrogen atoms obtained after the final cycle of refinement. The hydrogen distances varied from 0.97 to 1.11 Å. Figure 2 shows the bond angles between non-hydrogen atoms, as well as the angles around the hydrogen-bonded hydrogens. It is obvious that the compound in question has the skeletal configuration assigned as **1** in the solid state, *i.e.*, it is the *Z* isomer. The crystal structure consists of pairs of molecules, mutually hydrogen-bonded from S to N14 *via* H142. Additionally, each molecule contains two intramolecular hydrogen bonds, one between N12 and N1 *via* H12, and the other between N14 and N11 *via* H141. The hydrogen-bonding information is given in Table 1.

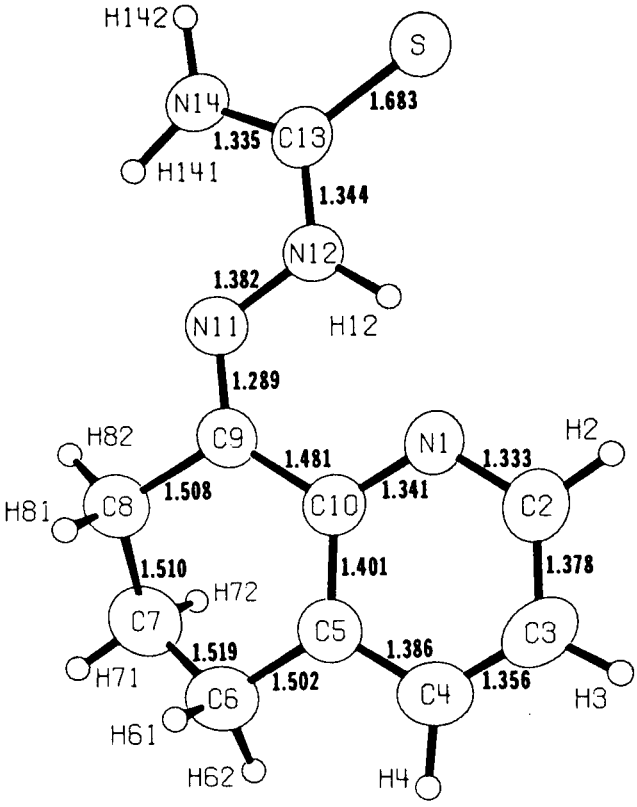


Figure 1. Intramolecular bond lengths (Ångstroms). The standard deviations average 0.006 A. The numbering of atoms is arbitrary.

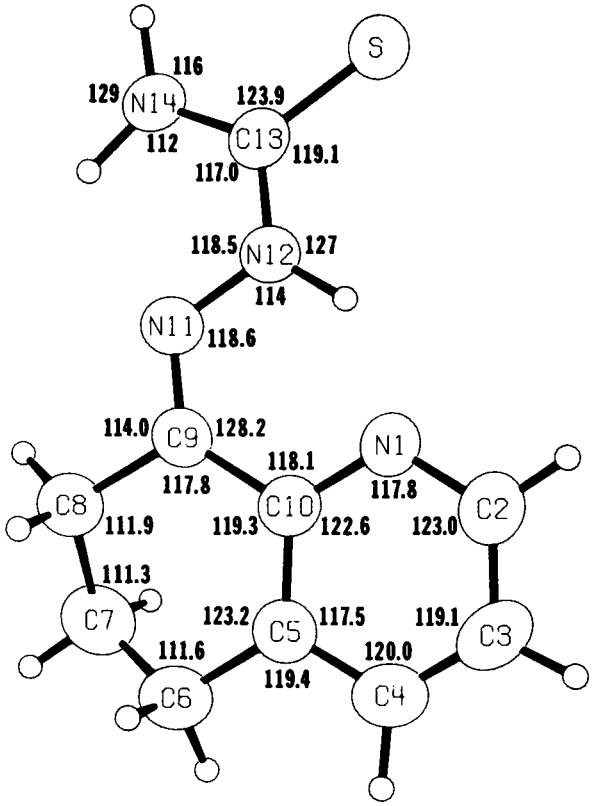


Figure 2. Intramolecular bond angles (degrees). The standard deviations of the angles between non-hydrogen atoms and of the angles involving hydrogens are 0.3° and 2°, respectively.

Table 1

Hydrogen-Bonding Information
(e.s.d.'s given in parentheses)

Atoms	Distance (Å)	Angle (°)
N14-H142	0.98 (3)	
N14--S	2.671 (3)	
H142--S	2.44 (3)	
N14-H142--S		166 (2)
N12-H12	1.03 (3)	
N12--N1	2.617 (4)	
H12--N1	1.74 (3)	
N12-H12--N1		139 (2)
N14-H141	1.07 (3)	
N14--N11	2.612 (5)	
H141--N11	2.06 (3)	
N14-H141--N11		108 (2)

Acknowledgment.

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REFERENCES AND NOTES

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(2) G. Germain, P. Main, and M. M. Woolfson, *Acta Cryst.*, **A27**, 368 (1971).